Imaging Composite Materials using X-Rays

Ersan Üstündag
Ames Laboratory and Department of Materials Science and Engineering, Iowa State University

Collaborators
Jay Hanan
Jet Propulsion Laboratory, California Institute of Technology

Yong Chu, Francesco de Carlo, Jon Almer, Ulrich Lienert and Dean Haeffner
Advanced Photon Source, Argonne National Laboratory

Irene Beyerlein
Theoretical Division, Los Alamos National Laboratory

Cev Noyan
Columbia University and IBM Research Division

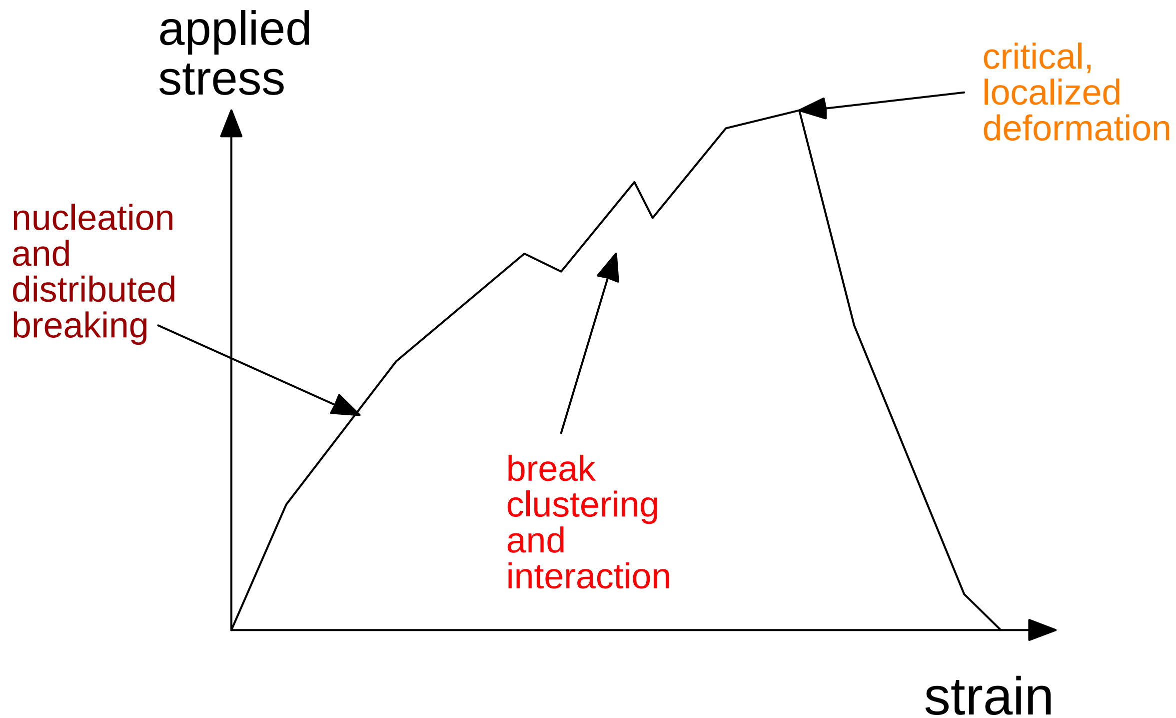
Supported by NSF (CAREER and MRSEC), DARPA, ARO and DOE

Outline

- **Introduction**
- **Metal matrix composites (Ti-SiC and Al-Al_2O_3)**
- **Ceramic matrix composites (SiC-SiC)**
- **Metallic glass matrix composites**
- **Cracks in ferroelectrics**

- ***Study of damage evolution in complex materials***

Fracture of a Fiber Composite under Tension

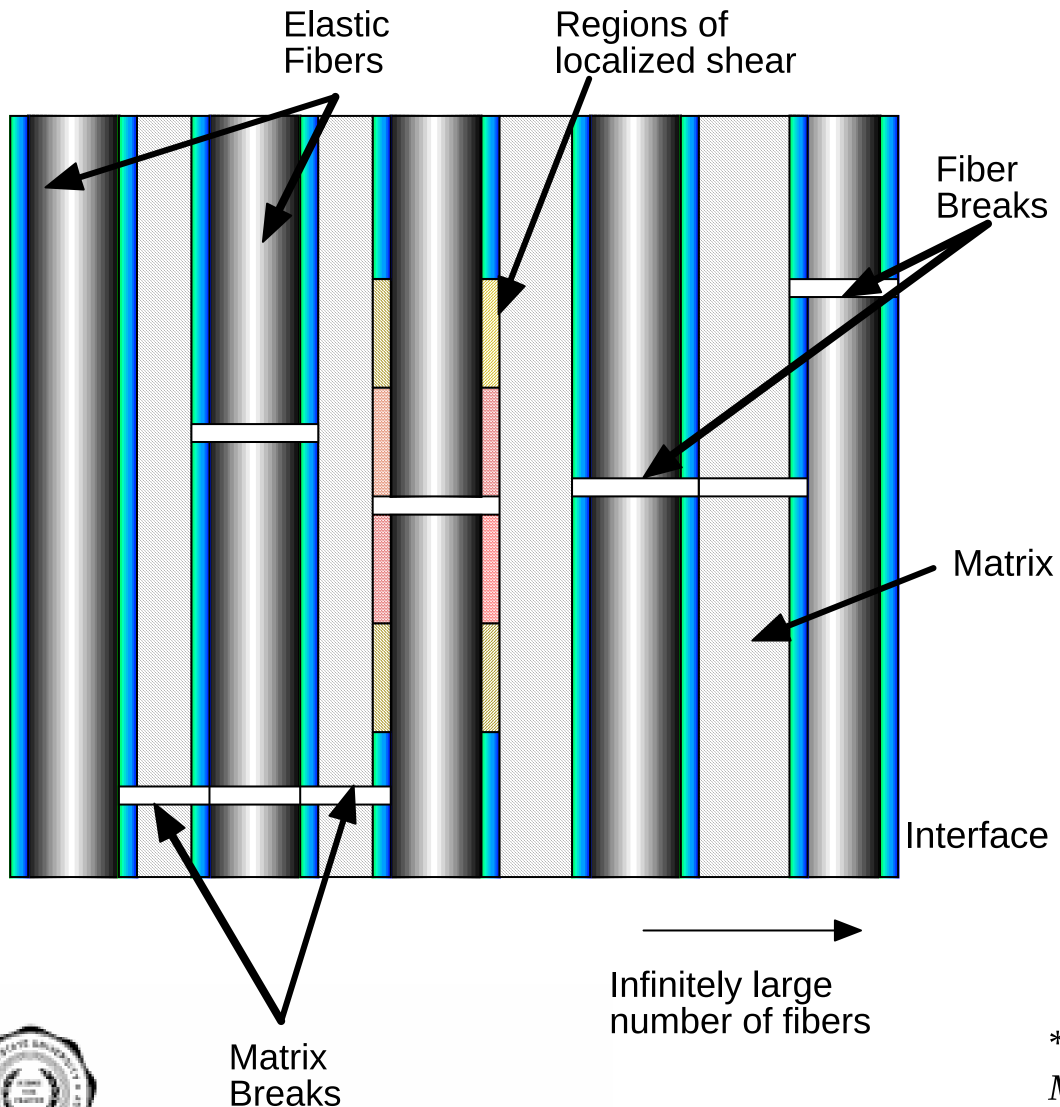


- **<u>Aim</u>: prediction of strength and lifetime**
- **<u>Need</u>: "realistic" constitutive laws and quantitative damage characterization**

<u>Complications</u>

- **Fabrication processes**
- **Inhomogeneous dislocation densities**
- **Changes in grain size**
- **Geometrical constraints**
- **Interface introduced with different properties**
- **Residual stresses**

Deformation of a Multi-Fiber Composite



Critical Parameters:

- Location and morphology of cracks
- Crack opening displacement
- Size of plastic zone
- Debond size at the interface
- Strain concentrations near cracks and debonds

* I.J. Beyerlein and C. M. Landis, *Mechanics of Materials*, 1999; 31: 331.

Motivation and Approach

- Little information about deformation and constitutive behavior of materials at <u>multiple length scales</u>.
- Need to link experimental data with rigorous micromechanics modeling.
- <u>Approach</u>: Use X-ray diffraction and imaging to investigate deformation in materials and complement it with modeling.
- Critical issues:
  - Need for model specimens
  - "High selectivity" of diffraction
  - Only elastic lattice strains are measured with diffraction
  - Lack of "realistic" constitutive laws to calculate stress and interpret diffraction data

Advantages of XRD

- **Non-destructive.**
- **Ability to distinguish different phases.**
- **Can measure elastic strain and texture.**
- **Simultaneous <u>strain</u> and <u>imaging</u> capability.**
- **Multi-scale: *nm* to *cm*.**
- **Deep penetration.**
- ***In-situ* experiment capability.**

$\Rightarrow$ Determination of *in-situ* constitutive behavior

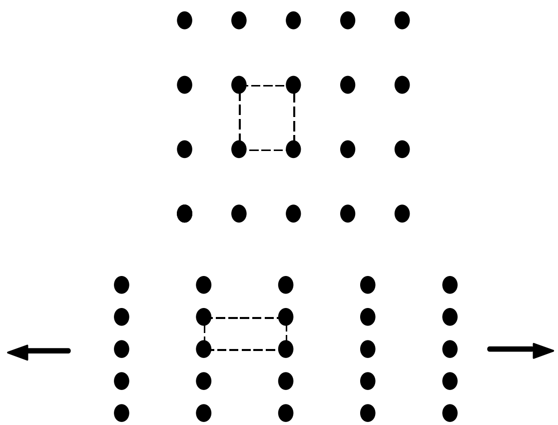
Bragg's law:

$$\lambda = 2d\sin\theta$$

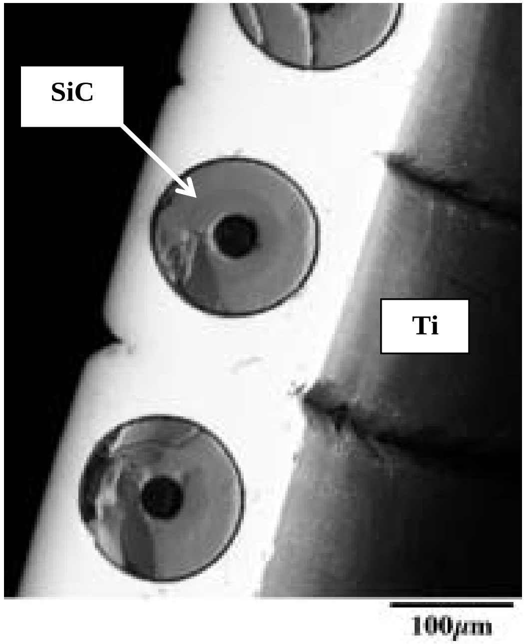
Differences in lattice spacing

$\Rightarrow$ Elastic lattice strain

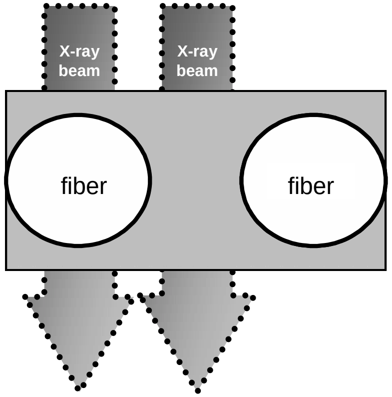
$$\varepsilon^{el}_{hkl} = \frac{d_{hkl} - d^{0}_{hkl}}{d^{0}_{hkl}} = \frac{d_{hkl}}{d^{0}_{hkl}} - 1$$

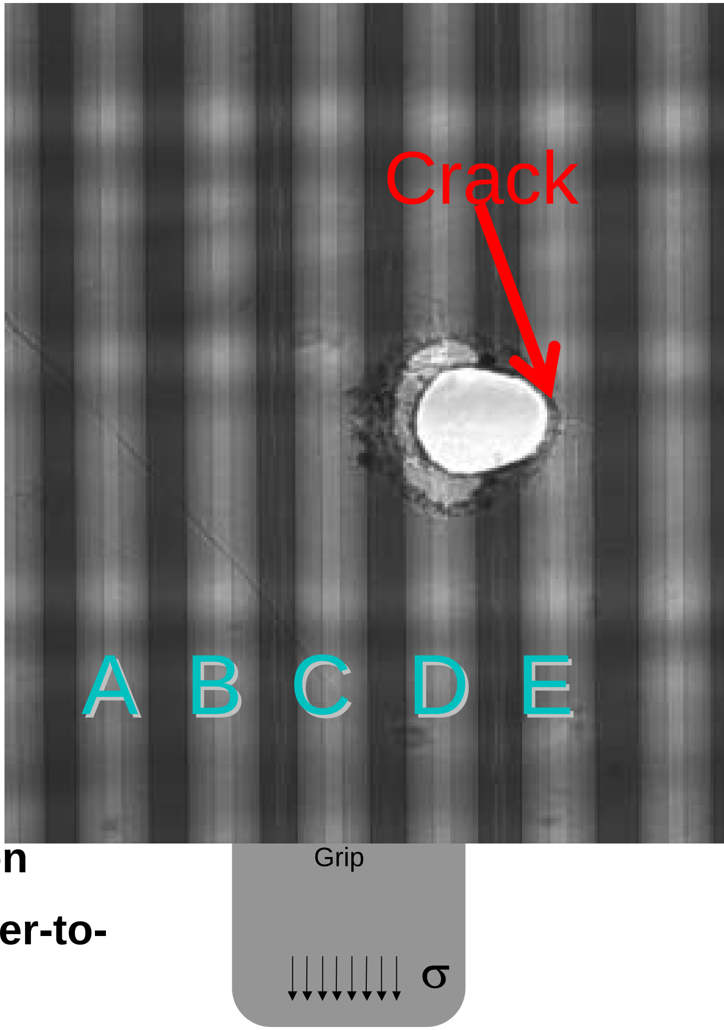


Model Composite: Ti-6Al-4V / SiC (SCS-6)



- Uniaxial tensile testing
- Damage evolution study using XRD (65 keV)
- Complete penetration
- 90 x 90 μm² spot size

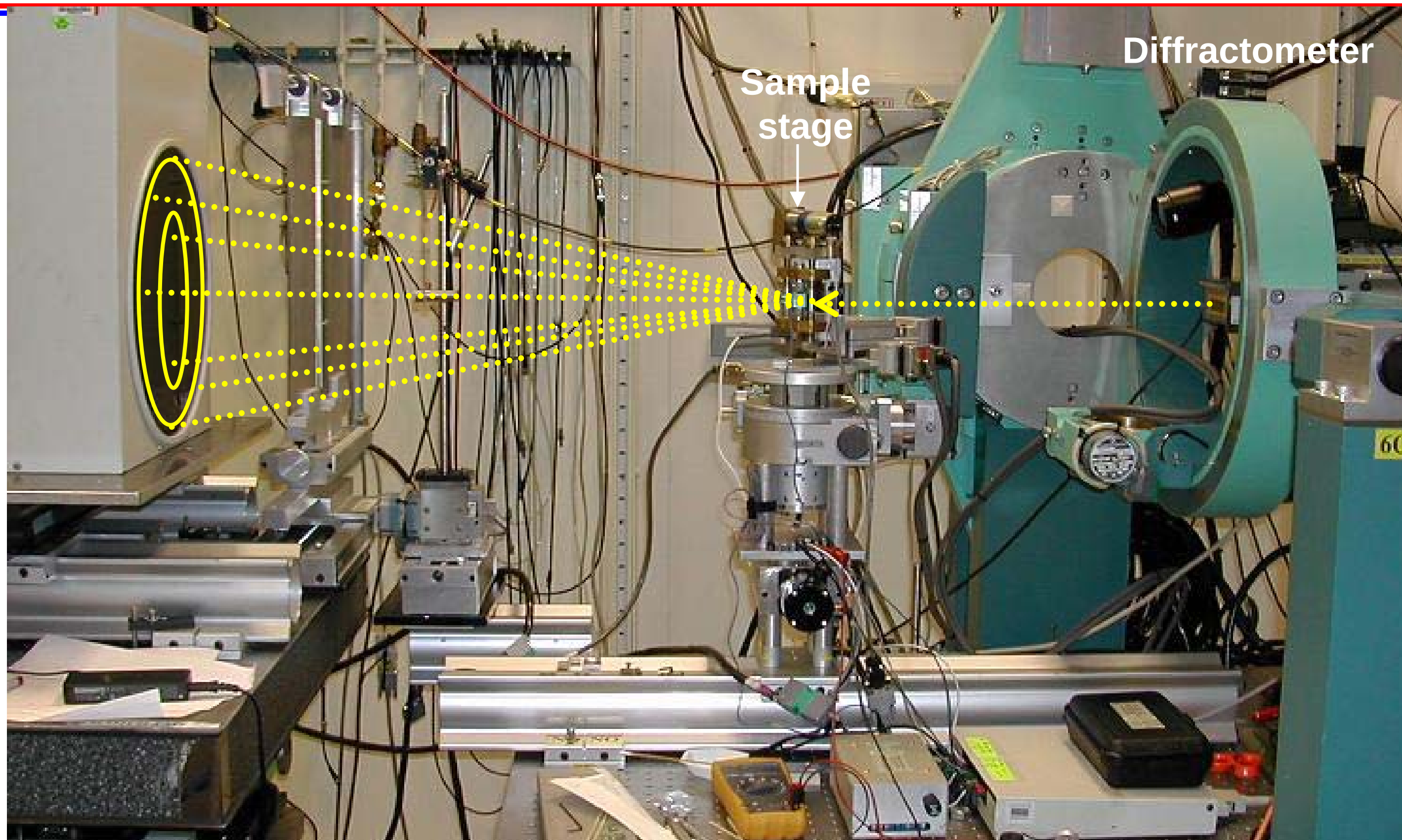


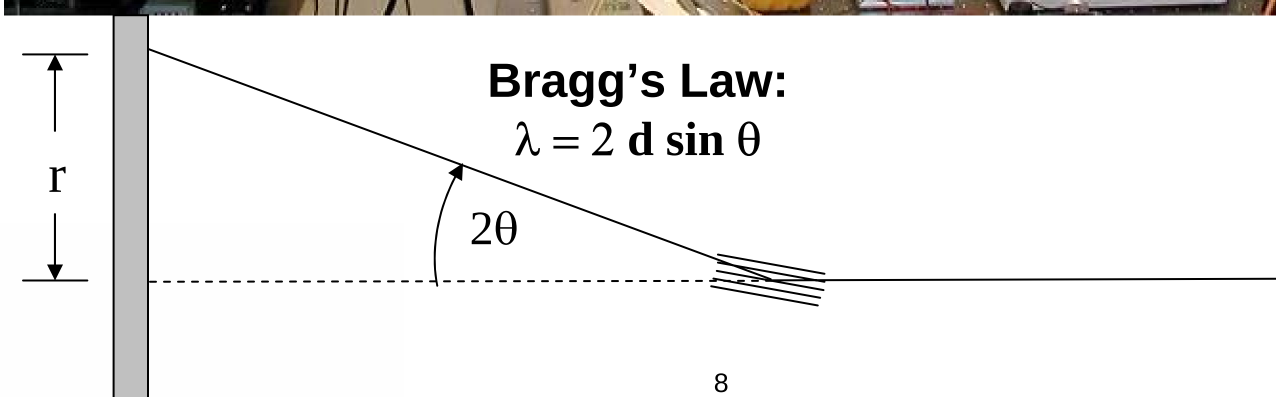


- Laminar composite: Ideal for model comparison
- 140 μm in diameter fibers; 240 μm average center-to-center distance
- 200 μm thick matrix
- Data collected with a digital image plate

High Energy 2-D XRD Experimental Setup

Digital image plate





Bragg's Law:

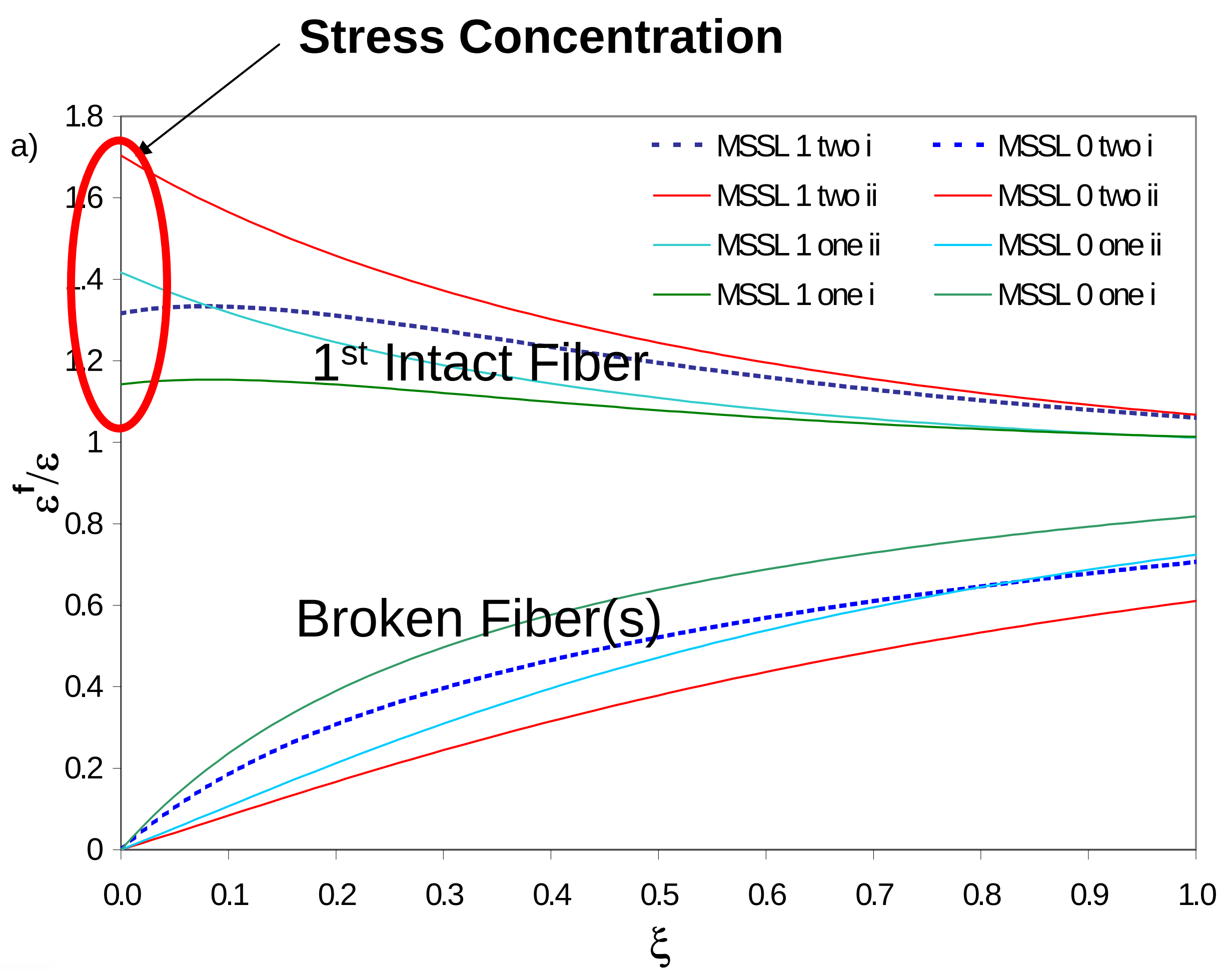
$$\lambda = 2\,\mathbf{d}\,\sin\theta$$

Matrix Stiffness Shear Lag (MSSL) Model Predictions

(i)-intact matrix

(ii)-broken matrix

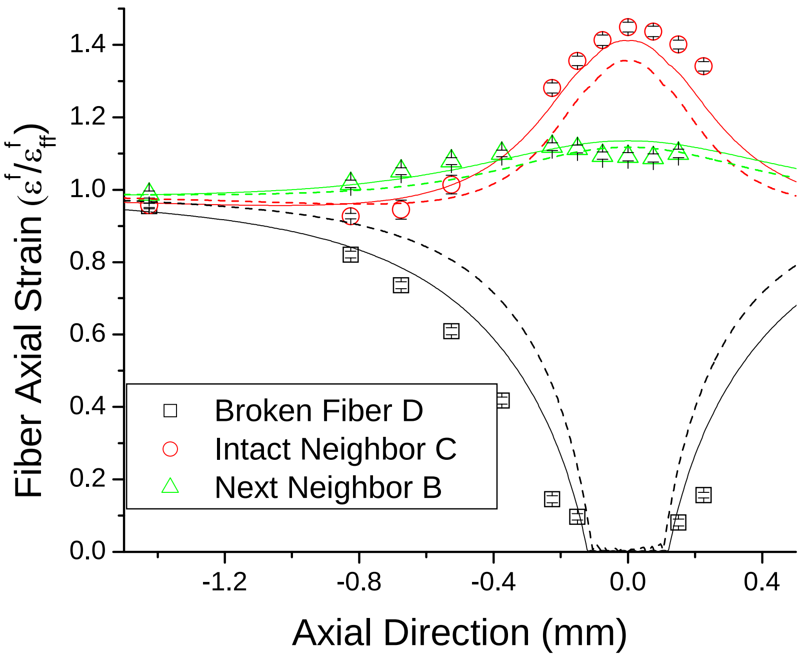
With $\rho = 0.289$ for both one and two broken fibers



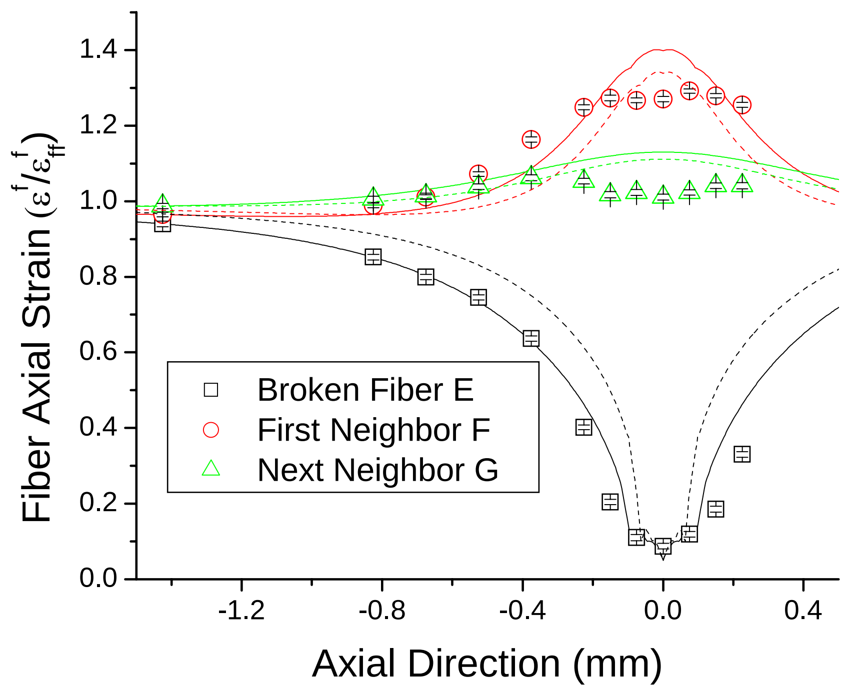
Unloading Strains in <u>Fibers</u> Compared to the MSSL Model

Ti-SiC

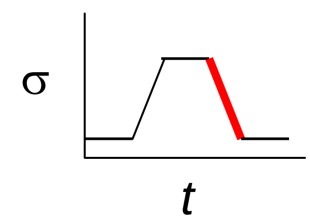
<u>Left Side of the Damage Zone:</u>



<u>Right Side of the Damage Zone:</u>



- **Good fit with 'intact matrix' case.**
- **Unloading strains were used due to plasticity in matrix.**
- **Right hand side data suggest interface debonding.**

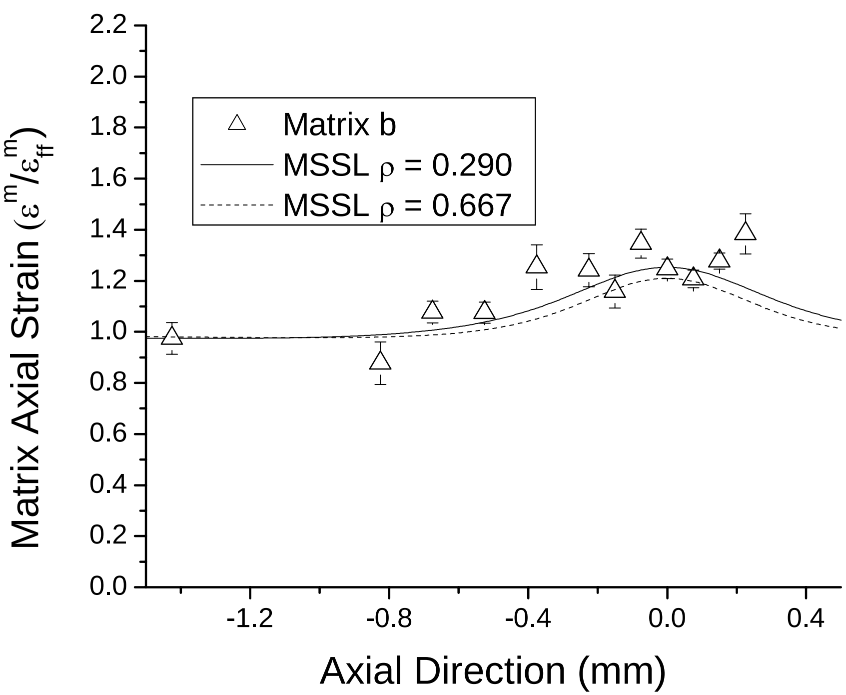


J. C. Hanan, E. Üstündag *et al*, *Acta Mater*. 51 [14], 4237-4248 (2003).

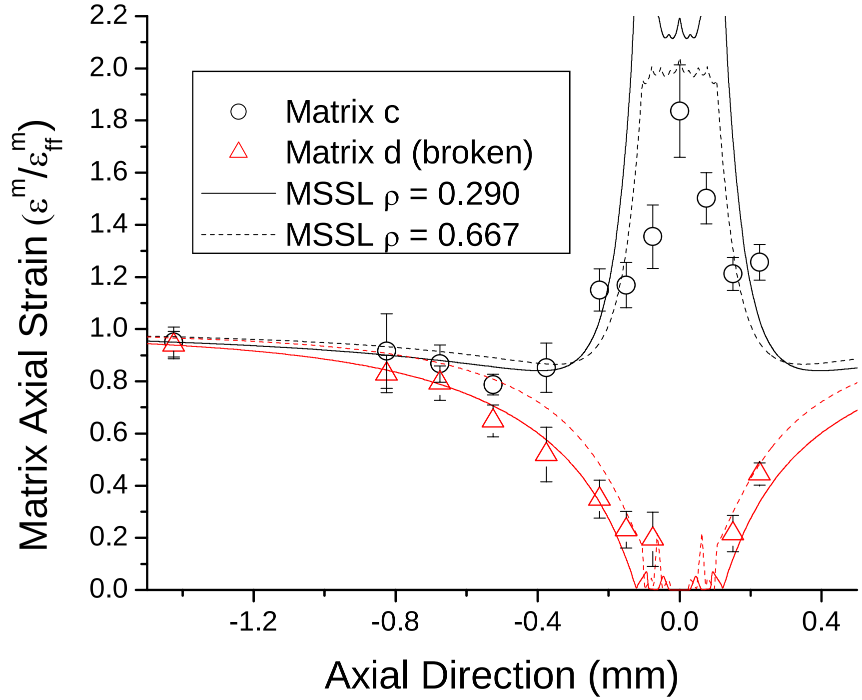
Unloading Strains in the Matrix Compared to the MSSL Model

Ti-SiC

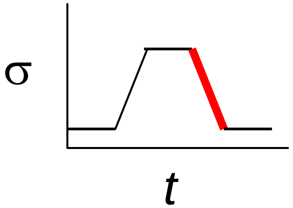
Matrix between two intact fibers:



Matrix around damage zone:



- Better fit with 'intact matrix' case.
- ρ = 0.290 appears to be a more realistic value.
- Matrix data comes from few grains.



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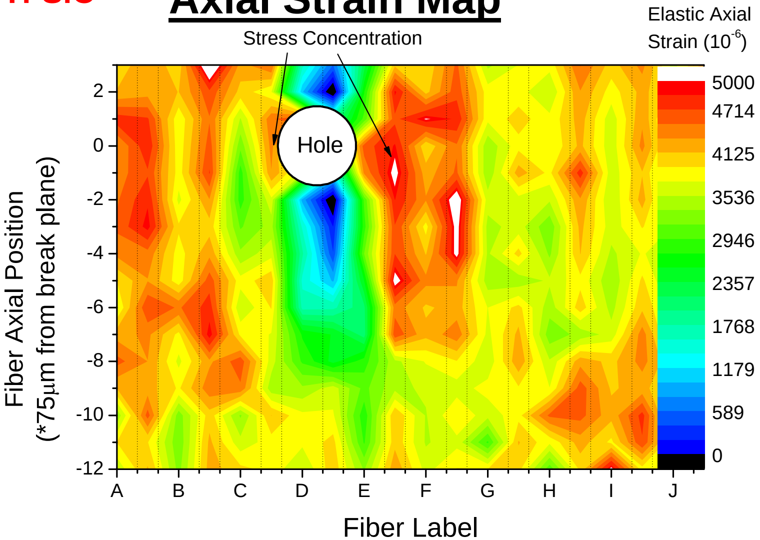
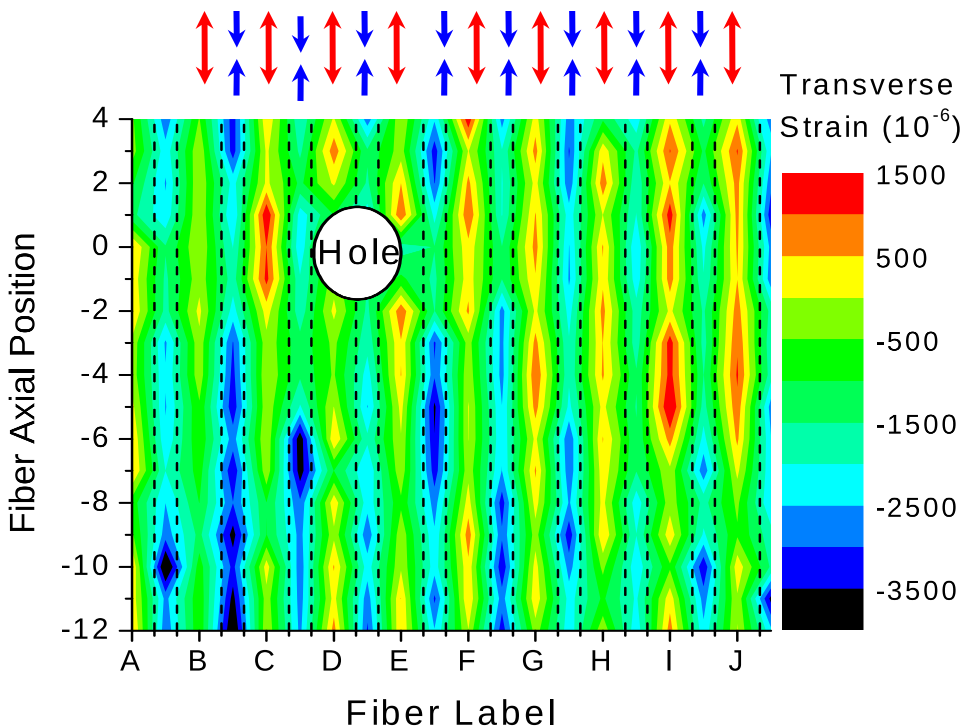
Matrix Strains using Image Plate

Ti-SiC

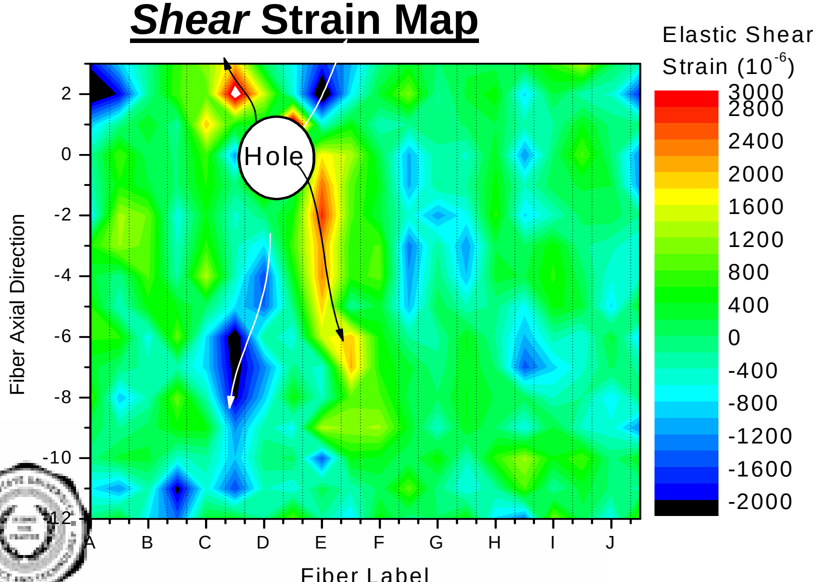
Axial Strain Map



Transverse Strain Map

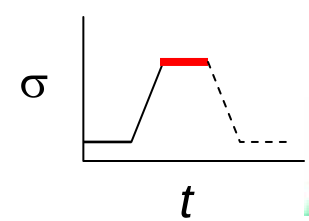


Shear Strain Map



- **Multi-axis strain data**
- **Significant strain concentrations in matrix**

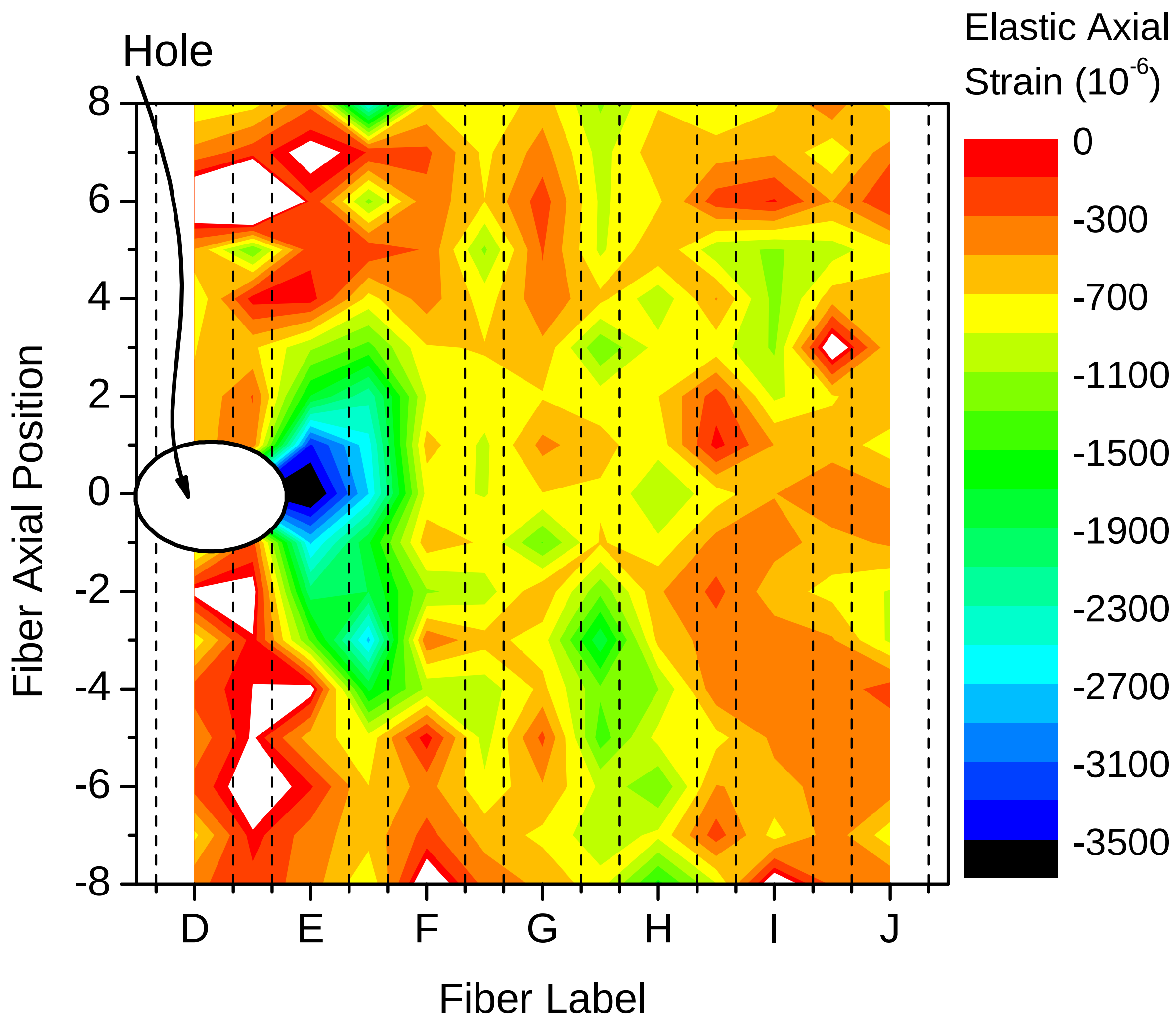
σ = 850 MPa

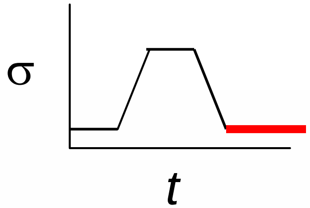


<u>Change</u> in Matrix ***Axial*** Residual Strain **due to Loading**

Ti-SiC

- **The compressive regions identify *<u>plastic</u>* deformation while loading the composite**

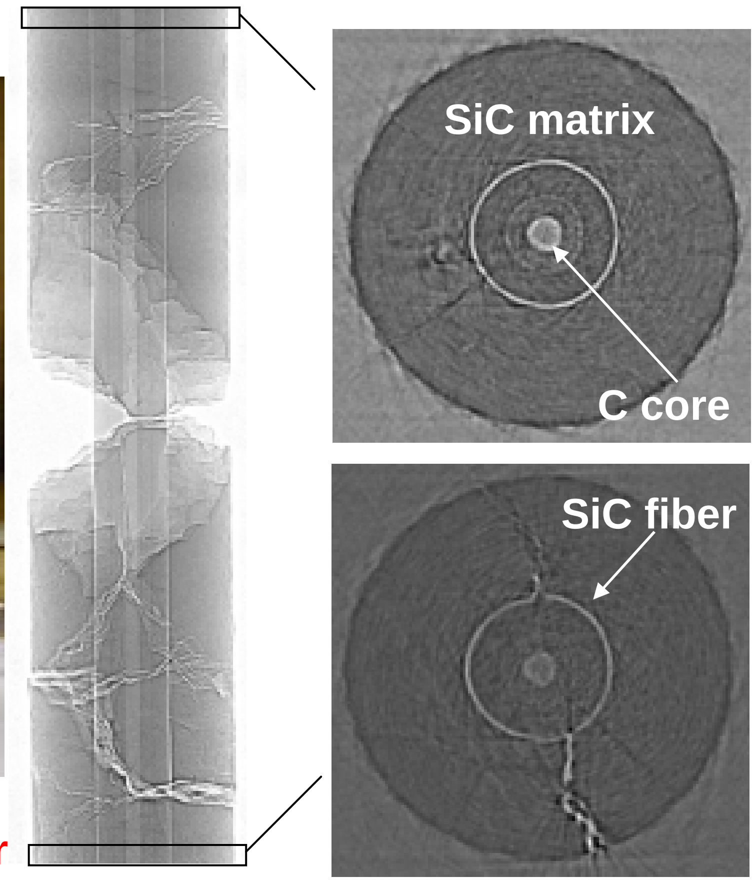




SiC/SiC Composite: *Microtomography*

- **Use of highly coherent X-ray beam (at APS).**
- **Contrast due to absorption and phase changes.**
- **Resolution ~ 1 µm.**
- **Ideal for damage evolution studies in CMCs:**
  - » **Multi-dimensional strain information**
  - » **Identification of cracks/debonds**

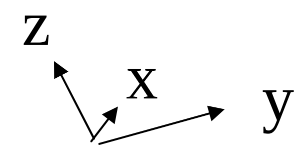
Special load frame for tomography

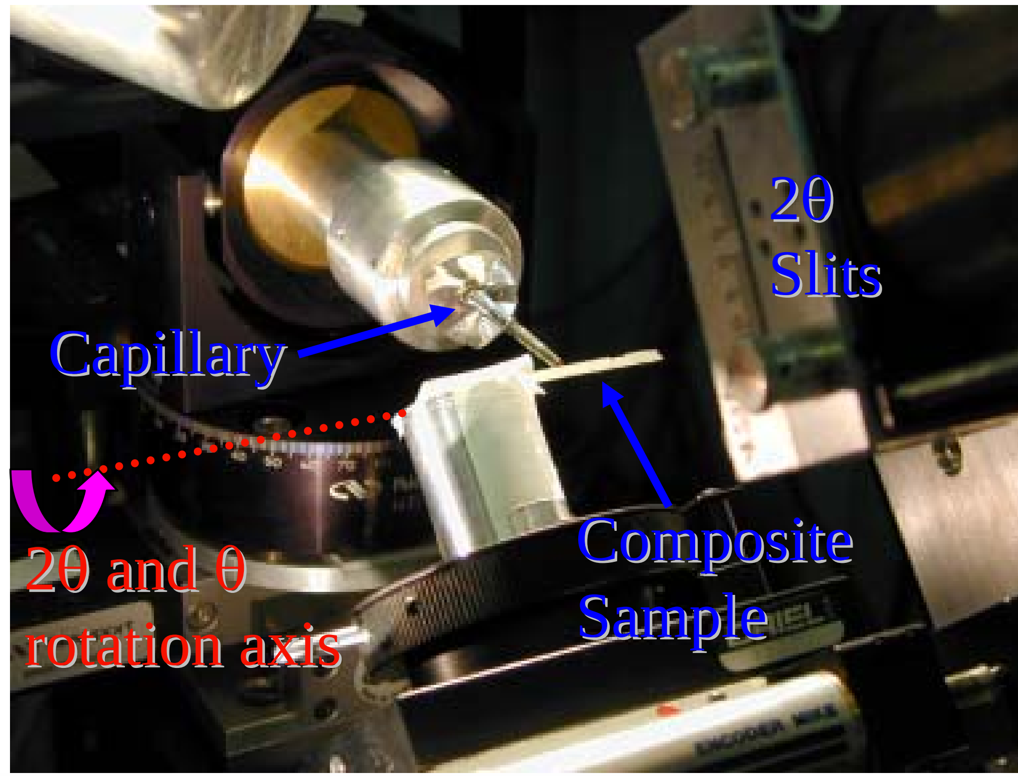


<u>Future</u>: Combined diffraction and tomography

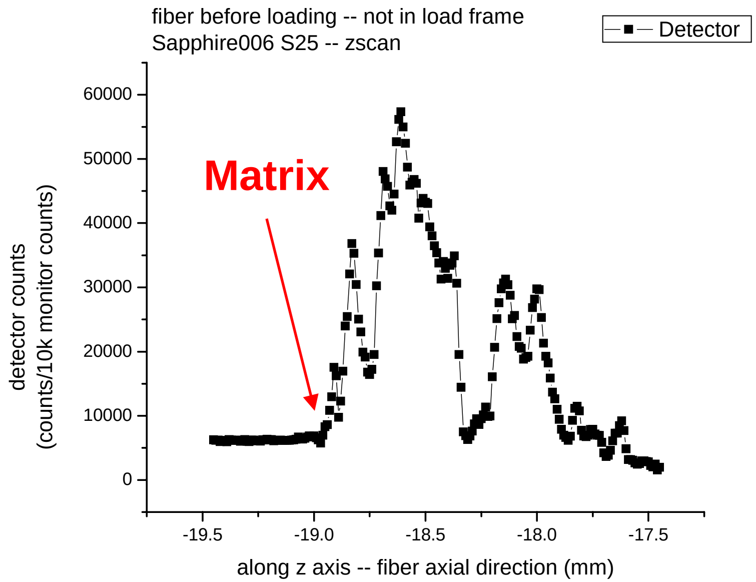
X-Ray Microtopography

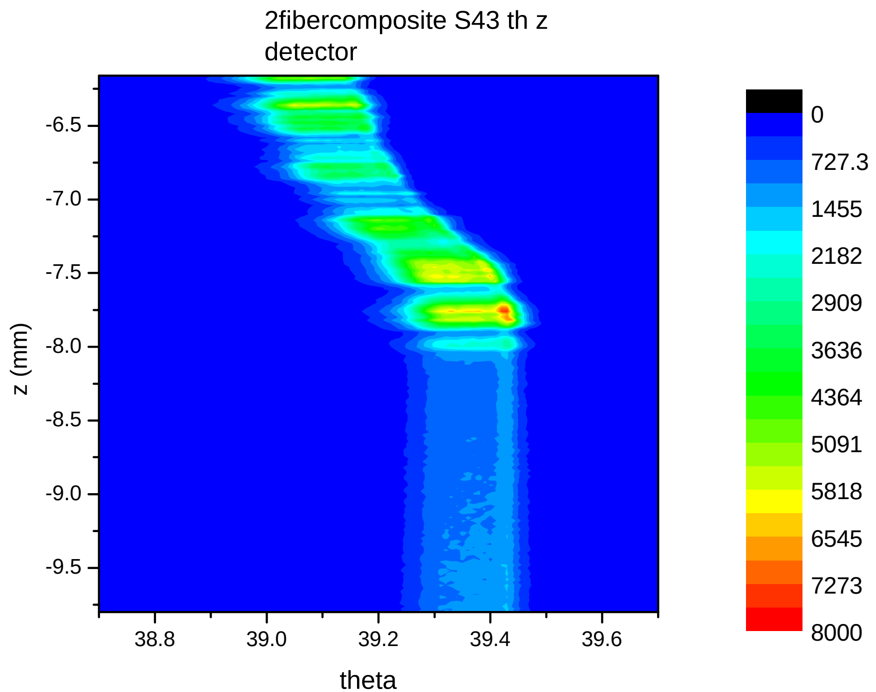
- National Synchrotron Light Source (BNL).
- Simultaneous diffraction, fluorescence and absorption.
- Microtopography provides information on the internal structure of composite:
  - matrix uniformity
  - interface integrity
  - fiber structure
- Spot size 2-10 μm using capillary.
- Tr
  a l

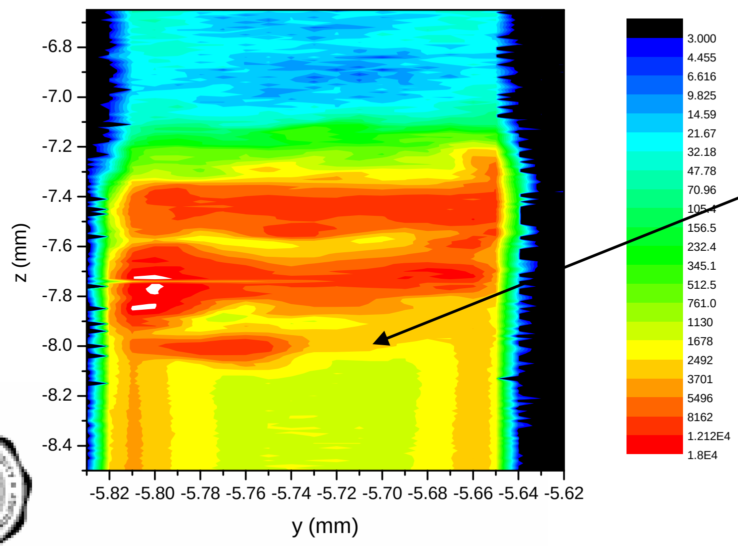




Effect of Matrix





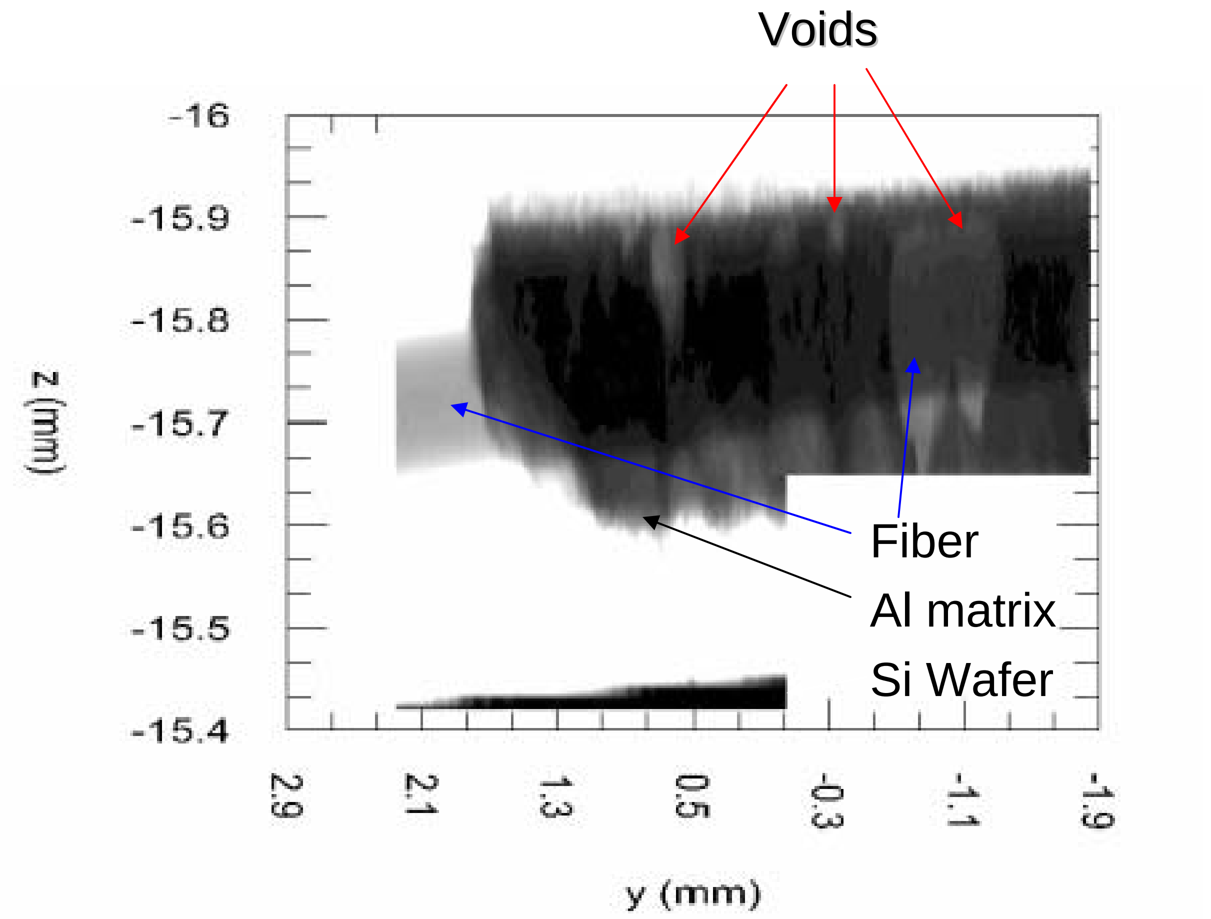


Matrix

"Bending" of fiber inside matrix

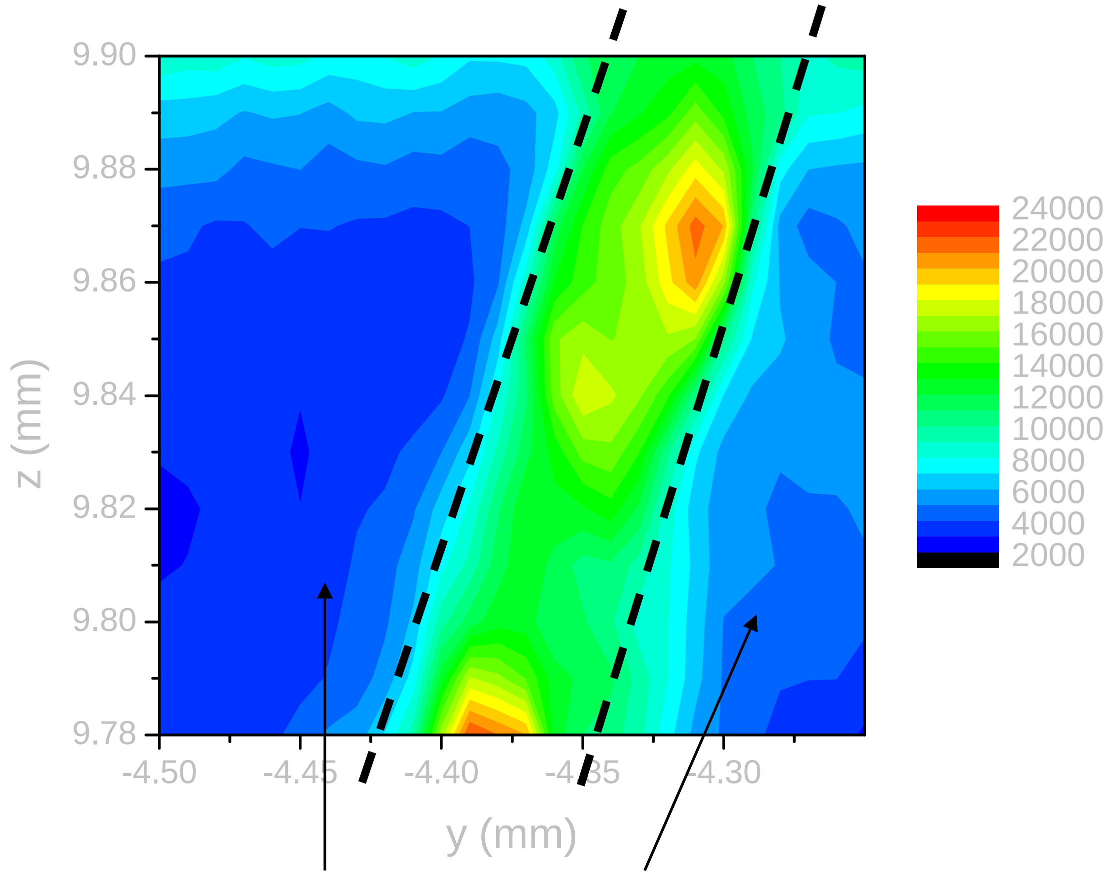
Transmission Profile

- $\theta = 0°$, $2\theta = 0°$.
- Some small voids are visible. The largest is located above the legend in the graph.
- The sample was fixed to the wafer on the opposite end of the sample from the one shown.



Radiograph of a Fiber Break

- Transmission reveals a 75 μm gap between the fibers.
- An *in-situ* measurement of a crack opening in a fractured fiber.

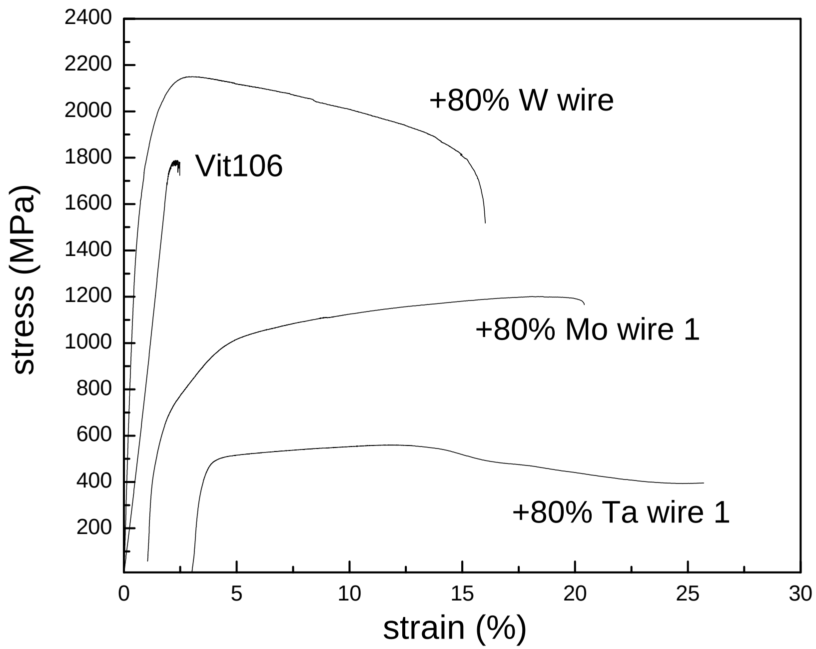


Broken fiber ends

Bulk Metallic Glass Matrix Composites

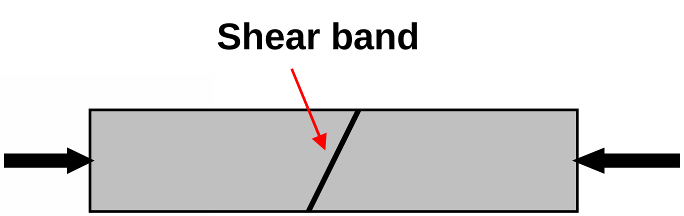
- Main problem with BMGs: **catastrophic failure** under unconstrained loading.
- Main **deformation mechanism** is via **shear bands** (at room T).
- Addition of reinforcements has been shown to increase damage tolerance and toughness.
- Critical questions:
  - What is the *in-situ* mechanical behavior of reinforcements?
  - How do reinforcements interact with shear bands?

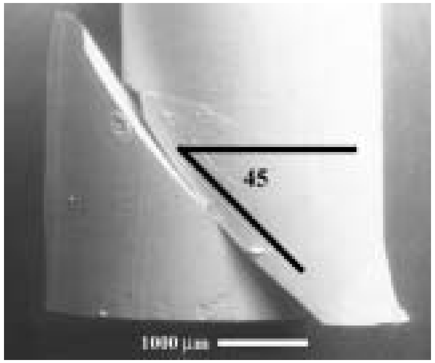
Compressive loading of fiber-reinforced BMG composites



Monolithic

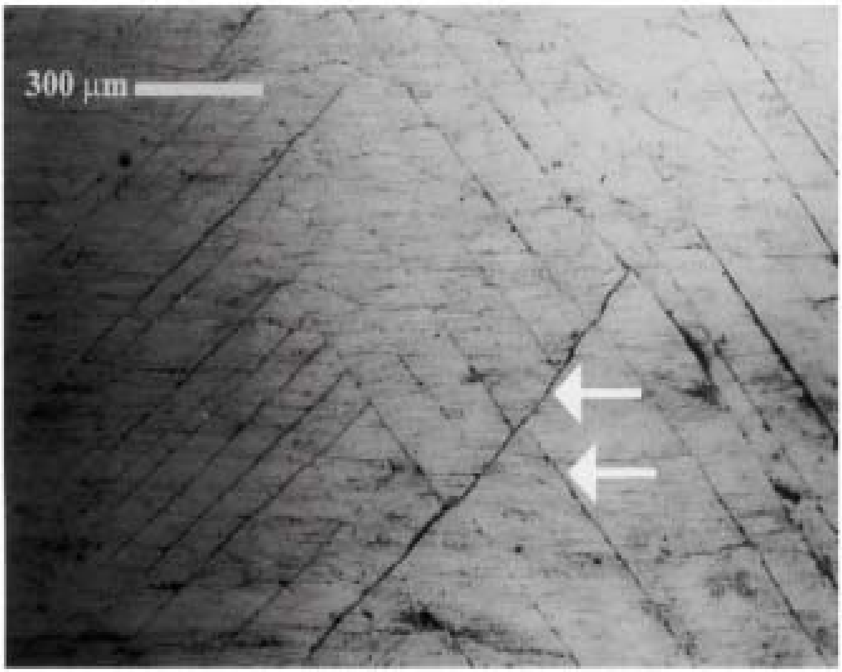
BMG



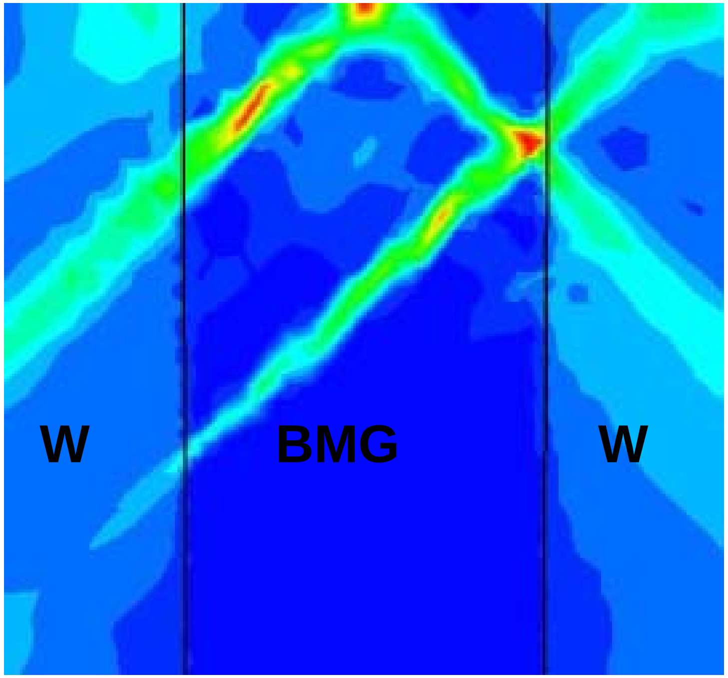


W-Fiber / BMG-Matrix Composites:
Damage Evolution

Multiple shear bands in 40% W-BMG composite



Modeling shear bands in W-BMG composites



- **BMG "yields" by multiple shear banding**
- **Slip initiates in W, transferred to BMG across interface**
- ***Details of micromechanics?***

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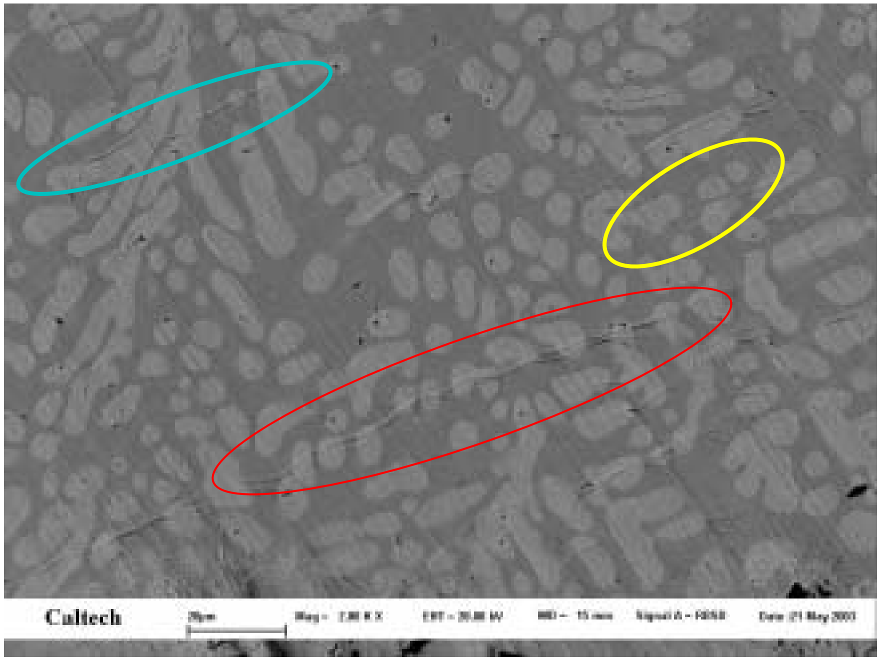
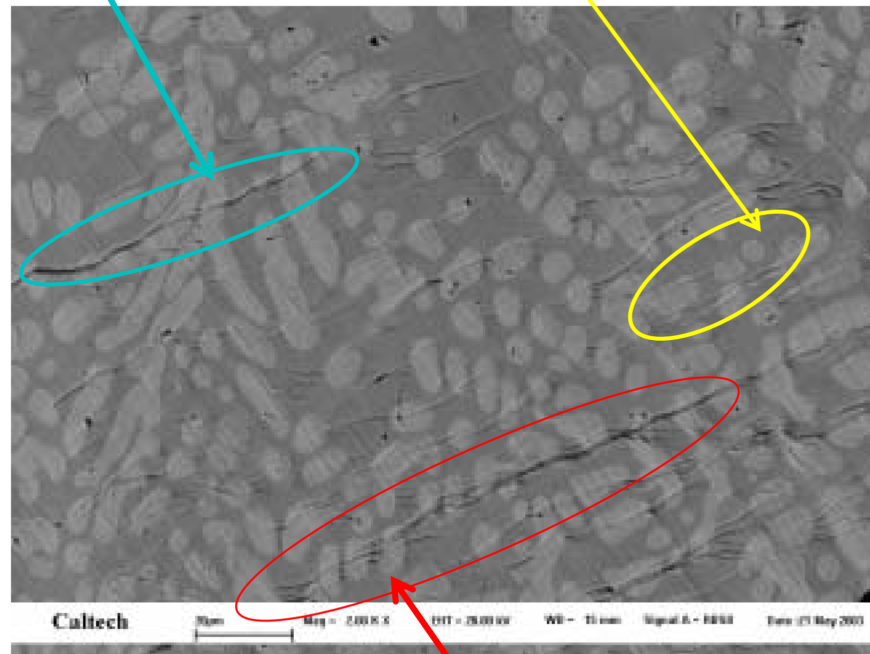
“β Phase” / BMG Composites:
SEM Investigations

Connect

Nucleate



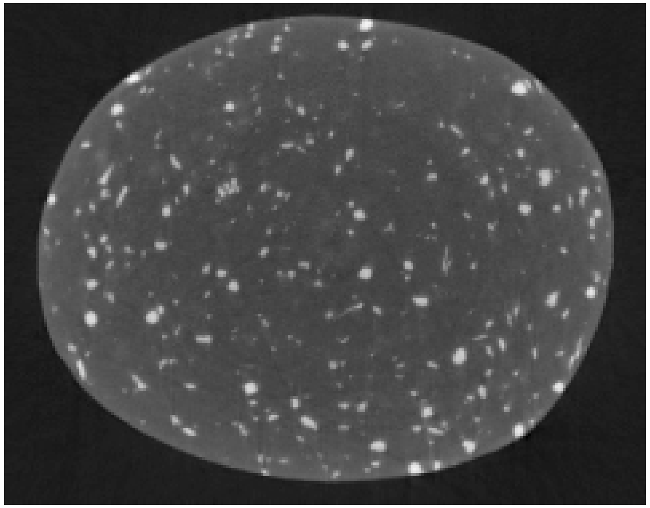


Slip

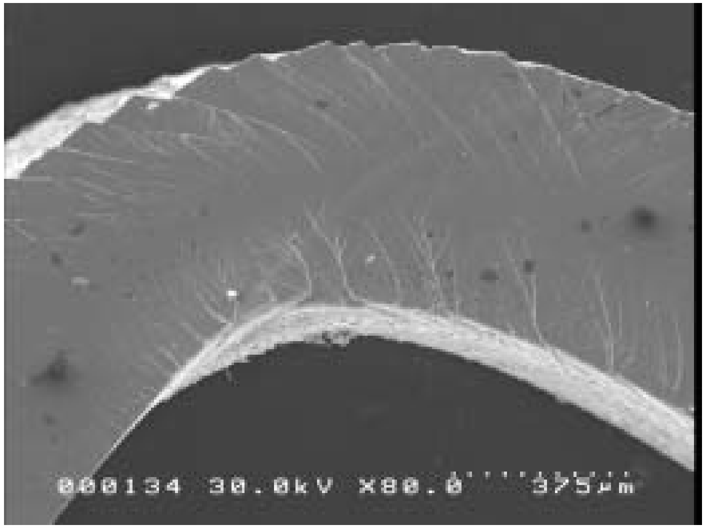
- Yielding initiates in dendrites.
- Shear bands connect precipitates.
- “Ideal” dendrite size and spacing?

BMG Matrix Composites: Present and Future Work

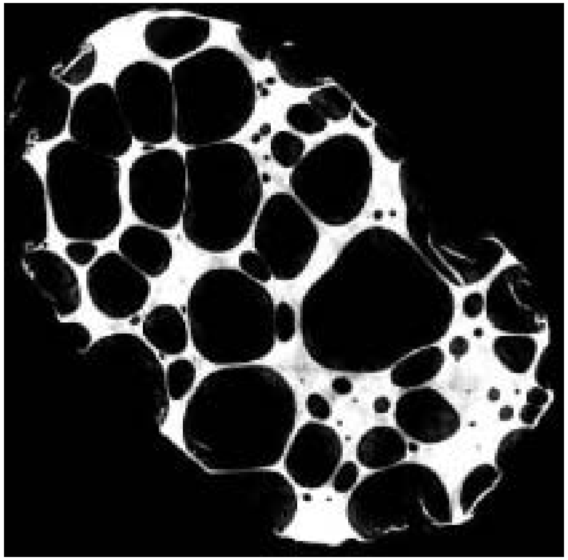
- **Deformation fields in model monoliths and composites:**
  - » **Laser speckle interferometry (2-D)**
  - » **X-ray tomography (3-D)**
  - » **Imaging of shear bands**
- **3-D FEM of deformation**



Tomograph of W-BMG composite



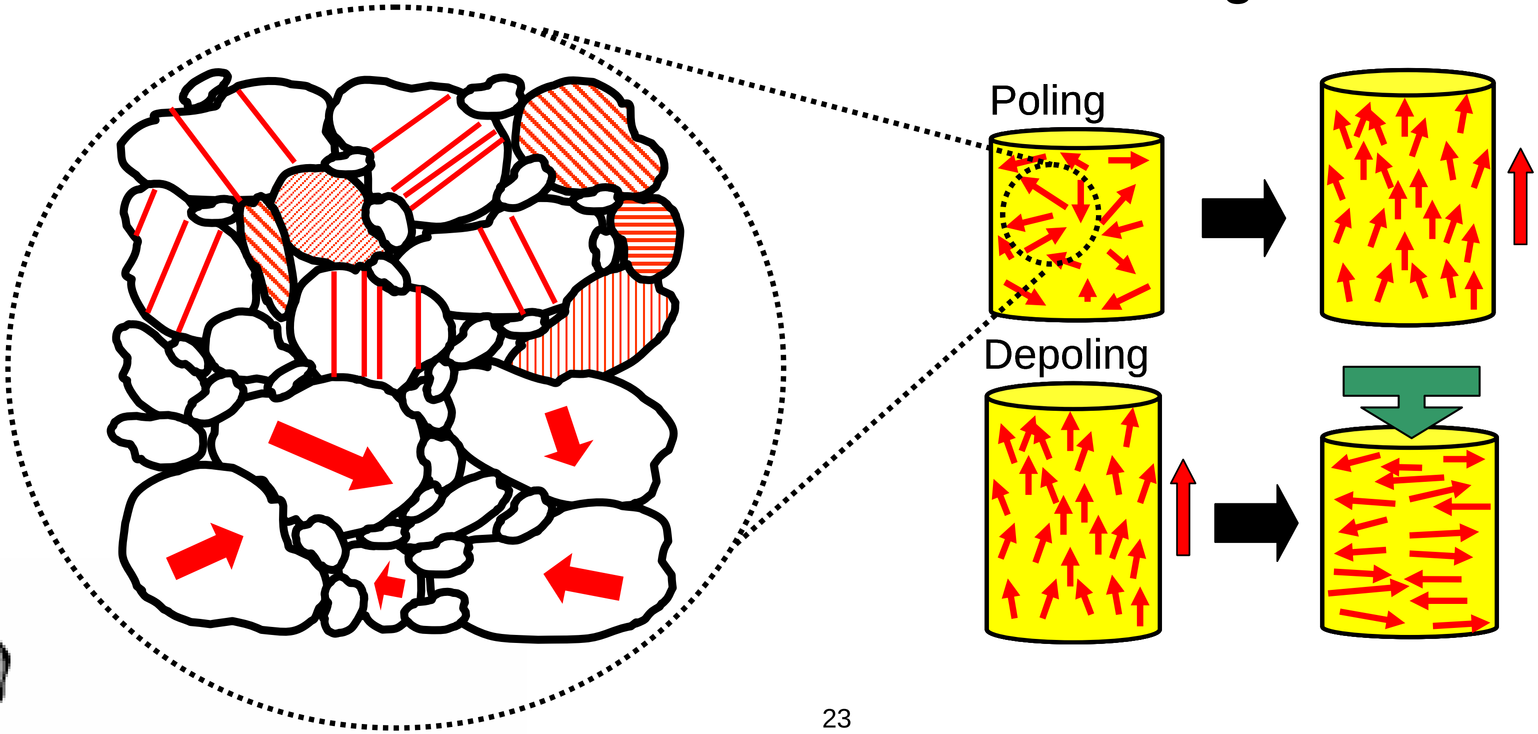
BMG under bending



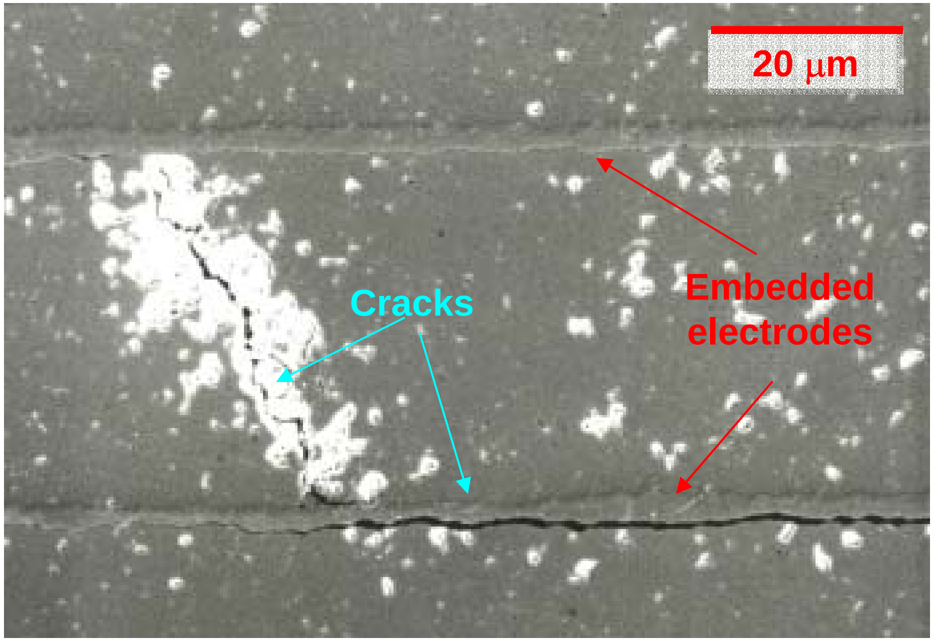
Tomograph of BMG foam

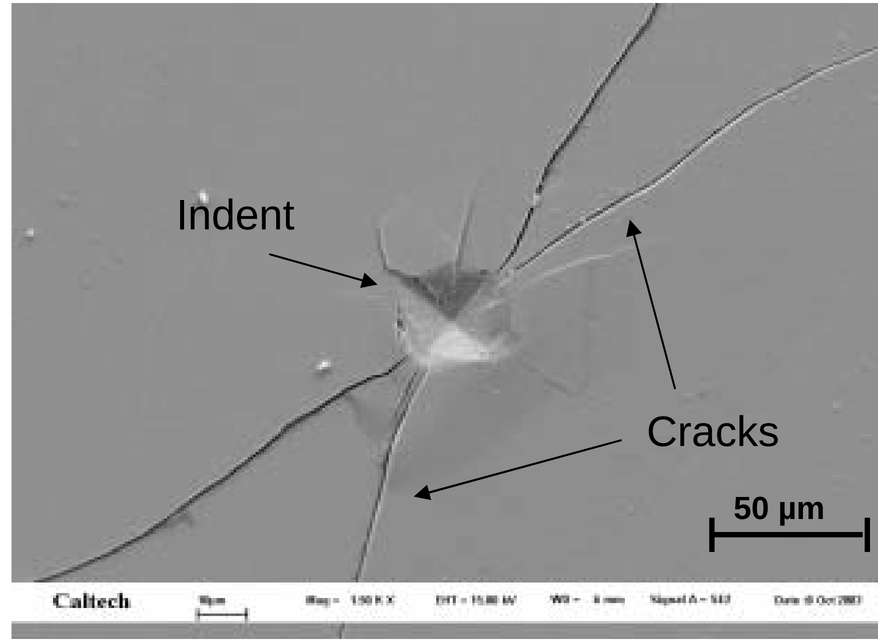
Ferroelectrics

- Grains within a polycrystal possess randomly oriented domains.
- Electrical poling is used to align a significant number of domains and produce a technologically viable ceramic material.
- Domain motion may be constrained by grain orientation and local boundary conditions.
- What are the details of inter-domain and inter-grain interactions?



Failure of Ferroelectrics





- **Low fracture toughness (K_{Ic} ~ 1 MPa.m$^{1/2}$)**
- **Indentation induced fracture mitigated by crystal poling direction; domain switching can impede crack propagation**
- **Domains interact with (and may induce) cracks**
- ***3-D morphology of cracks?***

Conclusions and Future Directions

- **X-ray imaging can revolutionize composites field**
- **<u>Grand challenge</u>: to monitor damage evolution in a composite in real time**
- **<u>Combined diffraction and imaging</u> capability needed**
- **Spatial resolution required: ~1 μm (or less)**
- **Real time data analysis is a necessity**
- **New ancillary equipment must be developed**
- **X-ray energy should be tunable within a wide range**

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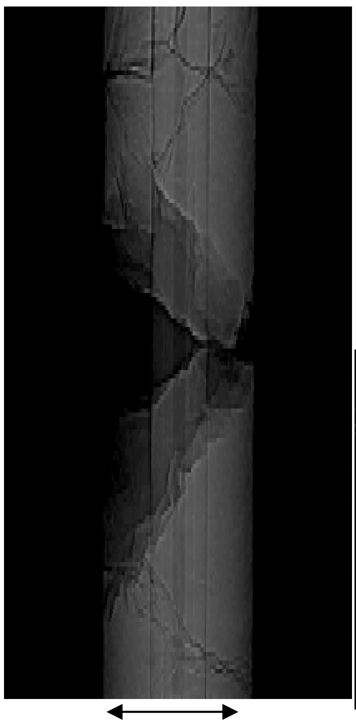
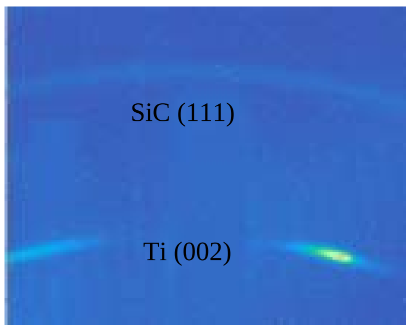
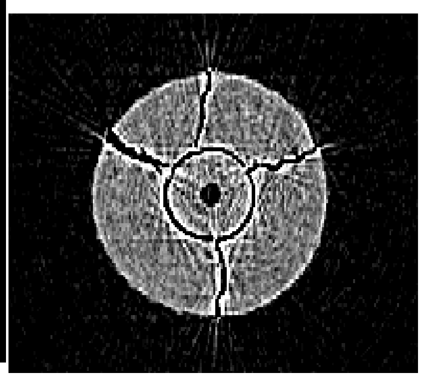
Development of *In-Situ* Tomo-Diffraction

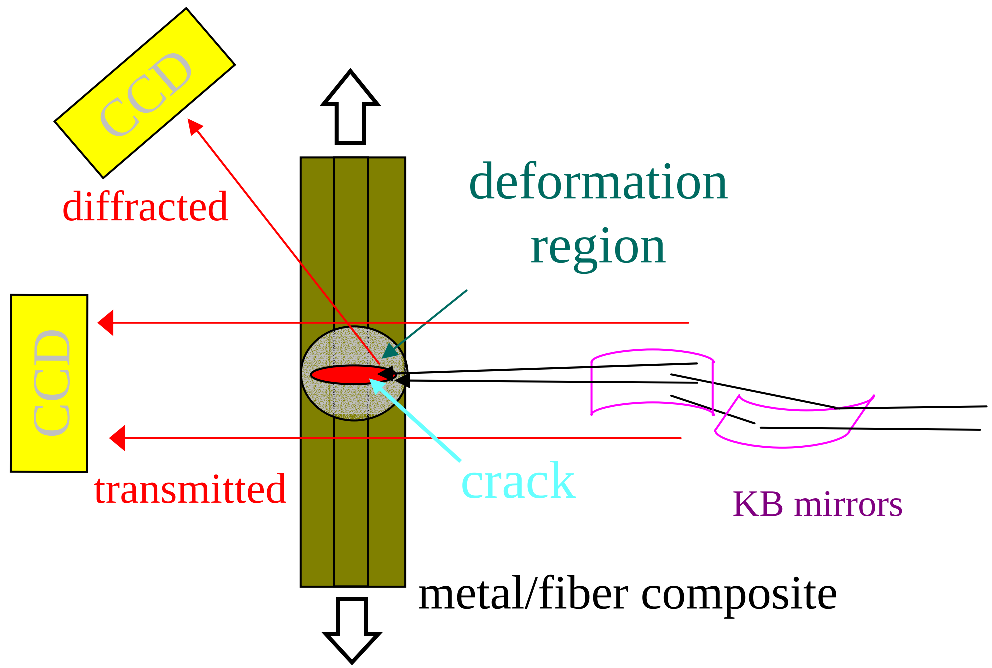
Goal: develop x-ray microscopy on both real space and reciprocal space, to investigate the deformation and fracture mechanics in engineering materials.

Strategy: dedicated comprehensive instrument development









“Development of an *In Situ* X-ray Tomography-Diffraction Technique”,
Y. S. Chu, F. De Carlo, J. D. Almer and D. C. Mancini, SRI, 2001
“Novel X-ray Diffraction Technique for Strain Measurements Using Area Detector”,
Y. S. Chu, F. De Carlo, and D. C. Mancini, Denver X-ray Conference, 2001